

## SRM 2492 RHEOMETER USER GUIDE FOR GAP VERIFICATION AND TEST PROTOCOL

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Measurements for SRM 2492 were made using a serrated parallel-plate rotational rheometer. The following user guide discusses the gap verification and experimental set-up for measurements made with SRM 2492. Other rheometer geometries and set-ups may be used and are described briefly in section 3 and in reference 1.

1. Gap between plates correction [1]: Prior to using a parallel-plate rotational rheometer, the gap between the plates needs to be verified and any correction factors applied. In such a rheometer, the viscosity calculated from the shear rate and shear stress should equal the nominal viscosity of a standard oil independently from the gap selected and whether the plates are serrated or not. Thus, the shear stress versus shear rate curves will overlap for various gaps and type of surface of the plates. If the curves do not overlap, a correction must be applied to account for the actual gap versus nominal gap, set by the rheometer controller. The following procedure may be used to determine the systematic error on the gap.

### Equipment and Material

- Serrated plates, top and bottom (smooth parallel plates can also be used, but the systematic error will be smaller than with serrated plates and usually, SRM 2492 should only be used with serrated plates)
- Standard oils with viscosity between 1 Pa·s and 30 Pa·s ( $\eta_i$ )

### Parameters

- Torque measured,  $M$  (N·m)
- Torque at the outer edge,  $T$  (N·m)
- Rotational speed,  $\Omega$  (rps or  $s^{-1}$ )
- Shear rate,  $\dot{\gamma}$  ( $s^{-1}$ )
- Gap between the parallel plates,  $h$  (mm)
- Plate diameter,  $d$  (mm) or plate radius,  $R$  (mm)
- Viscosity of the standard oil,  $\eta_i$  (Pa·s)
- Measured viscosity,  $\eta_x$  (Pa·s)
- Normalized viscosity (ratio of measured to standard viscosity),  $\eta_x/\eta_i$

*(Ensure that  $\Omega$  and  $M$  are recorded by the instrument in the correct units to use the equations below.)*

### Procedure

- Rheometer Protocol: shear rate range from 1  $s^{-1}$  to 50  $s^{-1}$  (other range could be used)
- For a nominal gap, measure and record torque  $M$  (N·m) and rotational speed  $\Omega$  ( $s^{-1}$ ) for at least 10 points during each sweep of increasing and decreasing shear rate.
- Repeat test for at least three nominal gaps, e.g., 0.3 mm, 0.5 mm, 1 mm.

### Interpretation of Results

If the curves of shear stress versus shear rate overlap, there is no systematic error on the gap (unlikely that there is no systematic error for serrated plates). If the curves do not overlap, a correction must be applied to account for the actual gap versus nominal gap. The overlap can be determined by calculating the slope of each curve, the average and standard deviation of the slopes could be calculated and the coefficient of variation should be less than 10%. The formula to determine the systematic error is shown in equation (5) and can be derived as follows. An excel worksheet is available that will perform the calculation for the user [2].

The viscosity of a Newtonian fluid in a parallel plate rotational rheometer can be calculated using equation (1).

$$\eta = \frac{2}{\pi R^3} \cdot \frac{dM}{d\dot{\gamma}} \quad (1)$$

The shear rate is calculated by

$$\dot{\gamma} = \frac{R}{h} \cdot 2\pi \cdot \Omega \quad (2)$$

Substituting into equation (1), and using the viscosity of the standard oil,

$$\eta_i = \frac{2}{\pi R^3} \cdot \frac{1}{2\pi \cdot \frac{R}{h}} \cdot \frac{dM}{d\Omega} \quad (3)$$

$$\eta_i = \frac{h}{\pi^2 R^4} \cdot \frac{dM}{d\Omega} \quad (4)$$

$dM/d\Omega$  can be calculated as the slope of the  $\Omega$  vs.  $M$  because the relationship between the rotational speed and the torque is linear. Solving for the gap, provides the corrected gap. The gap correction is the difference between the gap calculated from equation (5) and the nominal gap used.

$$h = \eta_i \pi^2 R^4 \cdot \frac{d\Omega}{dM} \quad (5)$$

This systematic error is calculated for each of selected nominal gap (in the example of Figure 1, nominal gaps of 0.3 mm, 0.6mm and 1 mm were selected). An average of the systematic gap error is calculated and it will be used as the systematic error gap for this rheometer.

An example of the normalized viscosity for gap measurements is shown in Figure 1. In this example, the measured normalized viscosity for nominal gap (shown as the blue diamonds – equation (1)) varies from the expected result (bold, red line). When the gap correction factor of 0.27 mm is applied (green triangles), the normalized viscosity is equal to 1 (or the calculated viscosity is equal to the certified value of the standard oil). Typically, the normalized viscosity should be between 0.95 and 1.05. More details on this method can be found in reference 1.

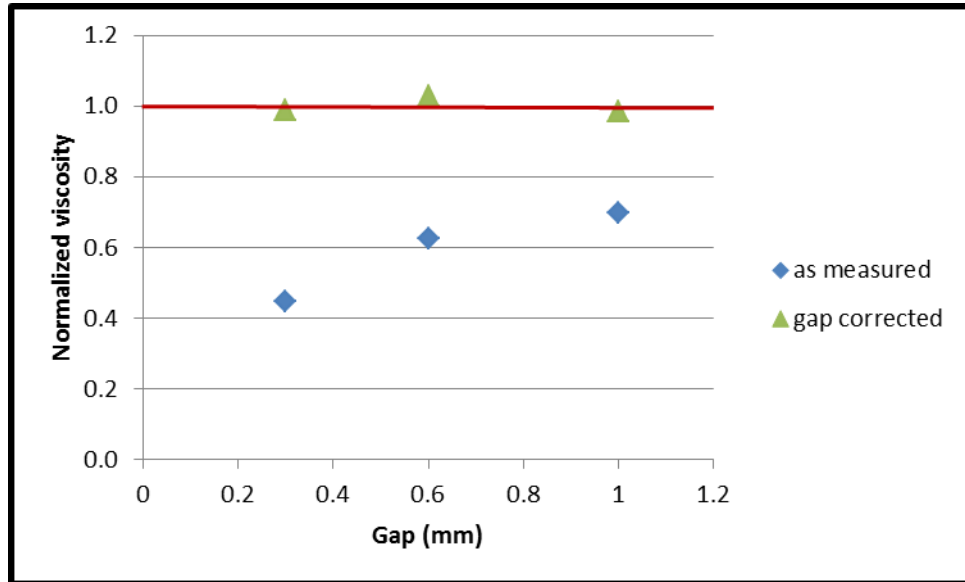


Figure 1: Gap correction. The normalized viscosity ( $\eta_x/\eta_i$ ) is the ratio of the measured viscosity to the nominal standard viscosity.

## 2. Test Protocol used for SRM 2492

### Experimental Set-up

- Temperature: 23 °C ± 1 °C at the rheometer
- Solvent trap: enclosure to ensure no evaporation of water during test. A wet sponge inside a plexiglass enclosure should be sufficient.
- Gap set to 0.6 mm  
(Note: The correction is applied to the results and is not taken into consideration during the test, see below.)

### Measurement Protocol Used for SRM 2492

- Constant shear rate ( $\dot{\gamma}$ ) at 0.01 s<sup>-1</sup> for 150 s
- Shear rate sweep increasing from 0.1 s<sup>-1</sup> to 50 s<sup>-1</sup> (10 steps)
- Shear rate sweep decreasing from 50 s<sup>-1</sup> to 0.1 s<sup>-1</sup> (20 steps)
- At each step, the shear rate was maintained until equilibrium (variation of the torque of less than 5%) of the torque was reached or 30 s, whichever came first.

### Calculation of Rheological Parameters from the Measurements

In the rheometer, rotational speed ( $\Omega$  in s<sup>-1</sup>) is controlled and torque is measured ( $M$  in N·m).

Computer software calculates the shear rate ( $\dot{\gamma}_R$ ) at the outer edge from the rotational speed using equation 2.

$$\dot{\gamma}_R = \frac{R}{h_c} \cdot 2\pi \cdot \Omega \quad (2)$$

- $R$  = radius of shear (mm)
- $h_c$  = corrected gap (nominal gap + the correction factor), i.e., distance between the plates (mm)
- $\Omega$  = speed of rotation of the top plate (s<sup>-1</sup>)

The shear stress ( $\tau$ ) can be calculated from the rotational speed and torque using the following.  $\frac{d \ln T}{d \ln \dot{\gamma}}$  is calculated by using a moving slope of every 3 points along the curve of T vs.  $\dot{\gamma}$ . The curve should be linear.

$$\tau = \frac{T}{2\pi R^3} \left( 3 + \frac{d \ln T}{d \ln \dot{\gamma}_R} \right) \quad (6)$$

This equation was used to determine the parameters provided in the SRM 2492 certificate.

### 3. Calibration for other geometries

Procedure:

- Load SRM 2492 in the rheometer
- Perform a sweep on increasing and decreasing rotational speeds
- At each speed, the torque should be recorded for 30 s or until it is stable (change of less than 5%), whichever happens first.
- Record the torque on at least 10 points in the decreasing rotational speed sweep.
- Plot the torque ( $M$ ) versus the rotational speed ( $\Omega$ ); calculate the slope ( $b$ ) and intercept ( $a$ ) of the linear relationship.
  - The slope,  $b$ , is proportional to the plastic viscosity  $\eta$  (in N·m·s)
  - The intercept,  $a$ , is proportional to the yield stress,  $\tau_0$  (in N·m)
- The correction factor ( $K_{\tau_0}$ ) for the geometry for yield stress,  $\tau_0$  is:

$$K_{\tau_0} = \frac{\tau_0}{a} \quad (7)$$

- The correction factor ( $K_\eta$ ) for the geometry for viscosity,  $\eta$  is:

$$K_\eta = \frac{\eta}{b} \quad (8)$$

These correction factors then can be used to compare data from different geometries as the Bingham rheological parameters would be in the same units.

### REFERENCES

- [1] Ferraris, C.F.; Geiker, M.; Martys, N.S.; Muzzatti, N.; *Parallel-plate Rheometer Calibration Using Oil and Lattice Boltzmann Simulation*; J. Adv. Concr. Technol., Vol. 5, Issue 3, pp. 363–371 (2007).
- [2] 2492 Gap Verification Worksheet, Excel spreadsheet available under the Data File tab on the details page, [https://www-s.nist.gov/srmors/view\\_detail.cfm?srm=2492](https://www-s.nist.gov/srmors/view_detail.cfm?srm=2492).